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[Document Name] Specification

[Title of the Invention] ACID OIL-IN-WATER TYPE EMULSION COMPOSITION

[Claim 1] An acid oil-in-water type emulsion composition comprising an oil phase containing at least 20 % by weight of diacylglycerol and 0.5 to 5.0 % by weight of a crystallization inhibitor.

[Claim 2] The acid oil-in-water type emulsion composition according to Claim 1, wherein the crystallization inhibitor is selected from polyglycerol fatty acid esters, sucrose fatty acid esters and sorbitan fatty acid esters.

[Claim 3] The acid oil-in-water type emulsion composition according to Claim 2, wherein the polyglycerol fatty acid esters are such that the average polymerization degree is 2 to 12, the number of carbon atoms in the fatty acid moiety is 12 to 22, and the degree of esterification is at least 70 %.

[Claim 4] The acid oil-in-water type emulsion composition according to Claim 2, wherein the sucrose fatty acid esters are such that the degree of esterification with a fatty acid having 12 to 22 carbon atoms is at least 50 %, and the hydroxyl groups not esterified with the above-mentioned fatty acids are acetylated.

[Claim 5] The acid oil-in-water type emulsion composition according to Claim 2, wherein the

sorbitan fatty acid esters are those in which the number of carbon atoms in the fatty acid composition is 12 to 22, and the HLB is lower than 3.

[Detailed Description of the Invention]

[0001]

[Technical Field to which the Invention Belongs]

The present invention relates to an acid oil-in-water type emulsion composition such as mayonnaise or dressing, which is excellent in emulsion stability even under low-temperature conditions such as in an refrigerator, good in appearance and flavor and useful as a diet or food for improving lipid metabolism.

[0002]

[Prior Art]

In recent years, it has been clarified that diacylglycerol (hereafter referred to as "DAG") has an obesity-preventing effect, an effect to prevent increase in weight, etc. (Japanese Patent Application Laid-Open No. 300828/1992, etc.), and it is attempted to incorporate this into various kinds of foods (Japanese Patent Registration No. 2848849, etc.).

[0003]

In the case of, particularly, an acid oil-in-water type emulsion composition such as mayonnaise or dressing, however, the low-temperature resistance has been insufficient in that a part of DAG in a raw oil

crystallizes under in-refrigerator conditions (-5 to 5°C) to cause demulsification (oil-off), and for mayonnaise, cracks were observed.

[0004]

[Problems Sought for Solution by the Invention]

It is therefore an object of the present invention to provide an acid oil-in-water type emulsion composition which is excellent in shelf stability at low temperatures though containing DAG at a high concentration, also good in appearance and flavor and useful as a diet or food improving lipid metabolism.

[0005]

[Means for the solution of the Problems]

The present inventors have found that an acid oil-in-water type emulsion composition satisfying the above requirements can be provided by containing a specified amount of a crystallization inhibitor in an oil phase containing DAG.

[0006]

The present invention thus provides an acid oil-in-water type emulsion composition comprising an oil phase containing at least 20 % by weight of diacylglycerol and 0.5 to 5.0 % by weight of a crystallization inhibitor.

[0007]

[Mode for Carrying out the Invention]

DAG used in the present invention is obtained by esterifying hydroxyl groups at 1- and 2-positions or 1- and 3-positions of glycerol with fatty acids. No particular limitation is imposed on the number of carbon atoms in fatty acid residues. However, it is preferably 8 to 24, particularly 16 to 22. The amount of unsaturated fatty acid residues is preferably at least 55%, more preferably at least 70%, most preferably at least 90% based on all the fatty acid residues. DAG is obtained by any known process such as ester exchange reaction of vegetable oil and/or animal oil with glycerol or esterification reaction of a fatty acid composition derived from the above oil with glycerol. The reaction method thereof may be either a chemical reaction method by the aid of alkali catalysts, for example, or a biochemical reaction method using an oil and/or fat hydrolase such as lipase. One or blends of such DAGs may be used. The content of DAG in the oil phase must be at least 20 % by weight, preferably at least 35 % by weight from the viewpoints of effectiveness as a diet and profitability. The oil phase may contain triacylglycerol, monoacylglycerol, free fatty acids and the like in addition to DAG. These may be derived from any of vegetable oils and/or animal oils.

[0008]

The crystallization inhibitor used in the

present invention is preferably selected from polyglycerol fatty acid esters, sucrose fatty acid esters and sorbitan fatty acid esters. The polyglycerol fatty acid esters are preferably those in which the average polymerization degree of glycerol is 2 to 12, the number of carbon atoms in the fatty acid moiety is 12 to 22, and the degree of esterification is at least 70 %. Furthermore, the HLB of polyglycerol fatty acid esters is preferably lower than 4.5, most preferably lower than 3.5. The sucrose fatty acid esters are preferably those in which the degree of esterification with fatty acids having 12 to 22 carbon atoms is at least 50 %, and the hydroxyl groups not esterified with above-mentioned fatty acids are acetylated. Furthermore, the sucrose fatty acid esters preferably have an HLB of lower than 3, more preferably lower than 2. The sorbitan fatty acid esters are preferably those in which the number of carbon atoms in the fatty acid moiety is 12 to 22, and the HLB is lower than 3, more preferably lower than 2.5. One or more of these crystallization inhibitors may be used, and the content thereof must be 0.5 to 5.0 % by weight, preferably 0.6 to 3.0 % by weight from the viewpoints of sufficient crystallization-inhibiting effect at low temperatures and flavor as food.

[0009]

The water phase in the acid oil-in-water type emulsion composition according to the present invention may contain water; vinegar; common salt; condiments such as sodium glutamate; saccharides such as sugar and starch syrup; seasonings such as *sake* and sweet *sake*; various kinds of vitamins; organic acids; spices; various kinds of vegetables or fruits; thickeners such as xanthan gum; dairy products such as milk; various kinds of fruit juices; proteins such as soybean protein; various kinds of phosphates; etc.

[0010]

A blending ratio (weight ratio) of the oil phase to the water phase in the acid oil-in-water type emulsion composition according to the present invention is preferably 10:90 to 80:20, more preferably 50:50 to 75:25.

[0011]

Yolk may be used in the acid oil-in-water type emulsion composition according to the present invention for the purpose of imparting emulsification and improving flavor. Yolk may be used in any form such as raw, frozen, powdery, salted or sugared. Furthermore, it may also be used in the form of the whole egg containing yolk. The content of the yolk in the acid oil-in-water type emulsion composition according to the present invention is preferably 5 to 20 % by weight, more preferably 7 to 17 % by weight,

most preferably 8 to 15 % by weight based on weight of raw yolk from the viewpoint of improvement in flavor.

[0012]

In the acid oil-in-water type emulsion composition according to the present invention, a proportion (hereafter referred to as "lyso proportion") of lysophospholipid to total phospholipids contained is preferably at least 15 %, more preferably at least 25 %, most preferably 29 to 60 % from the viewpoints of shelf stability, appearance and flavor. A part or the whole of the lysophospholipid is preferably derived yolk and/or soybean, most preferably from yolk. Furthermore, a part or the whole of the lysophospholipid is preferably enzyme-treated yolk. The enzyme used in the enzyme treatment of yolk is preferably esterase, lipase or phospholipase, more preferably lipase or phospholipase, most preferably phospholipase. Among various kinds of phospholipases, phospholipase A, i.e., phospholipase A1 or A2 is most preferred.

[0013]

Examples of production forms of the acid oil-in-water type emulsion composition according to the present invention include those defined by Japanese Agricultural Standard (JAS) as dressing, semi-solid dressing, emulsified liquid dressing, mayonnaise,

salad dressing and French dressing, but the forms are not particularly limited to these products. All kinds of products, widely accepted as mayonnaise and dressing, correspond thereto.

[0014]

The acid oil-in-water type emulsion composition according to the present invention can be produced in accordance with, for example, the following process. Oil components such as DAG and the crystallization inhibitor are first mixed to prepare an oil phase. Yolk and other water-soluble raw materials are then mixed to prepare a water phase. The oil phase is added to the water phase, and the mixture is homogenized following preliminary emulsification if needed, whereby an acid oil-in-water type emulsion composition can be obtained. Examples of homogenizers include high-pressure homogenizers such as Mountaingorin and Microfluidizer, ultrasonic emulsifiers, colloid mills, agitating homomixers, and Milder.

[0015]

The acid oil-in-water type emulsion composition according to the present invention may be used in the same manner as usual mayonnaise, dressing or the like.

[0016]

[Examples]

In the following examples, the HLB of the

crystallization inhibitors was calculated out in accordance with the Griffin's empirical equation for polyglycerol fatty acid esters and sorbitan fatty acid esters. For sucrose fatty acid esters, emulsification method was used to determine HLB.

[0017]

Referential Example 1:

Preparation of Oil Composition 1

Lipozyme IM (product of Novo Nordisk Industry Co.) was added to a mixture of rapeseed oil fatty acid (650 parts by weight) and glycerol (107 parts by weight) and subjected to esterification reaction at 40°C for 5 hours under 7 hPa. The resultant reaction mixture was then subjected to molecular distillation (235°C, 0.07 hPa). The distillate thus obtained was then bleached, washed with water and deodorized at 235°C for 2 hours to obtain Oil Composition 1 having a composition shown in Table 1.

[0018]

Referential Example 2:

Preparation of Oil Composition 2

A mixture of soybean oil fatty acid (650 parts by weight), after the content of saturated fatty acids had been reduced by winterization, glycerol (107 parts by weight) and calcium hydroxide (2 parts by weight) was reacted at 230°C for 0.5 hours under nitrogen gas atmosphere, the reaction mixture was

allowed to stand for 12 hours to separate the glycerol phase, and then the resultant oil phase (oil composition) was washed with a 50 % by weight aqueous solution of citric acid whose proportion was 2 parts by weight to 100 parts by weight of the oil phase followed by centrifugation to afford oil composition. The resultant oil composition was then subjected to molecular distillation (235°C, 0.07 hPa), and the distillate thus obtained was then bleached, washed with water and deodorized at 235°C for 2 hours to obtain Oil Composition 2 having a composition shown in Table 1.

[0019]

[Table 1]

		Oil composition	
		1	2
Composition of *1 oil	Triacylglycerol	13.8	13.5
	Diacylglycerol	84.7	85.2
	Monoacylglycerol	1.2	1.0
	Free fatty acid	0.3	0.3
Fatty acid *2 composition	C16: 0	4.2	2.6
	C18: 0	1.9	0.7
	C18: 1	58.3	30.0
	C18: 2	21.3	57.5
	C18: 3	10.7	6.8
	C20: 0	0.7	1.5
	C20: 1	1.8	0.4
	C22: 0	0.2	0.1
	C22: 1	0.8	0.1

*1: Analyzed by gas chromatography after silylation.

*2: Analyzed by gas chromatography after methylation (Number of carbon atoms in fatty acid: number of carbon-carbon double bonds).

[0020]

Referential Example 3:

Preparation 1 of enzyme-treated yolk

A yolk solution (750 g) containing common salt (10 % by weight) was mixed with water (250 g). After the resultant mixture was preheated at 50°C for 10 minutes, Phospholipase A2 (enzymatic activity: 10,000

IU/mL) was added to the mixture. The proportion of phospholipase to the yolk solution was 100 ppm. Reaction was carried out for 3 to 5 hours, thereby obtaining Enzymolysed Yolk Solution 1. Incidentally, the lyso proportion was calculated out in accordance with the following method.

[0021]

The reaction product was first extracted repeatedly with a mixed solvent (3:1) of chloroform:methanol to obtain a lipid mixture. The resultant lipid mixture was subjected to thin layer chromatography to separate various kinds of phospholipids by two-dimensional thin layer chromatography of the first dimension: chloroform-methanol-water (65:25:49) and the second dimension: butanol-acetic acid-water (60:20:20). The amounts of the separated phospholipids collected were measured by means of a commercially available measuring kit (permanganate ashing method, Phospholipid Test Wako; product of Wako Pure Chemical Industries, Ltd.) to calculate out contents of lysophospholipids and total phospholipids. The lyso proportion (%) was defined as "(total amount of phosphorus in lysophospholipid fraction/total amount of phosphorus in all phospholipid fractions) x 100".

[0022]

Referential Example 4:

Preparation 2 of enzyme-treated yolk

Twice amount of water by volume was added to Enzymolysed Yolk Solution 1 obtained in Referential Example 3 and stirred. The mixture was then spray-dried under air (inlet air temperature =f 170°C, outlet air temperature = 70°C) to obtain enzyme-treated yolk powder.

[0023]

Test Example 1 (Examples 1 to 8 and Comparative Examples 1 to 3):

Oil phases and water phases of their corresponding compositions shown in Table 2 were prepared in a conventional manner. After each of the oil phases was added to its corresponding water phase while stirring the water phase to preliminarily emulsify the oil phase, the emulsion was homogenized by a colloid mill (5,000 rpm, clearance: 0.35 mm) to prepare mayonnaise having an average emulsified droplet diameter of 2.5 to 3.5 μm. The mayonnaise was charged into a mayonnaise tube (100 g).

[0024]

The respective mayonnaise samples thus obtained were stored for 1 month at -5 to 5°C and then left to stand at room temperature for 3 hours. Thereafter, the appearances and physical properties thereof were evaluated by 6 panelists in accordance with the following respective evaluation standards. The

average values of these evaluations are shown in Table 2.

[0025]

Evaluation standard:

(1) Appearance:

The appearance of each mayonnaise sample charged into a tube was visually evaluated.

3: Good;

2: Signs of separation were observed;

1: Obvious separation took place.

[0026]

(2) Physical property

The mayonnaise sample squeezed out of the tube was visually evaluated

3: Good;

2: Signs of roughening on the surface and/or water separation were observed;

1: Obvious roughening on the surface and/or water separation took place.

[0027]

[Table 2]

(% by weight)

			Example								Comparative Example		
			1	2	3	4	5	6	7	8	1	2	3
Water phase		Common salt	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30
		Sucrose	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
		Condiment	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40
		Mustard powder	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
		Thickener *1	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20	0.20
		Enzyme-treated yolk	20.0	20.0	20.0	20.0	20.0	20.0	-	-	20.0	20.0	20.0
		Enzyme-treated yolk powder	-	-	-	-	-	-	7.40	3.70	-	-	-
		Yolk	-	-	-	-	-	-	-	7.50	-	-	-
		10% Brewed vinegar	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00	6.00
		Water	1.90	1.90	1.90	1.90	1.90	1.90	14.50	10.70	1.90	1.90	1.90
Oil phase		Oil Composition 1	20.7	20.5	19.5	20.5	20.5	34.5	20.5	20.5	21.0	35.0	20.8
		Oil Composition 2	48.7	48.5	47.5	48.5	48.5	34.5	48.5	48.5	49.0	35.0	48.8
		Polyglycerol fatty acid ester *2	0.6	1.0	3.0	-	-	1.0	1.0	1.0	-	-	0.40
		Sorbitan fatty acid ester *3	-	-	-	1.0	-	-	-	-	-	-	-
		Sucrose fatty acid ester *4	-	-	-	-	1.0	-	-	-	-	-	-
Lyso proportion (%)			45	45	45	45	45	50	55	55	45	50	50
Evaluation	5℃	Appearance	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
		Physical property	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
	0℃	Appearance	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	2.0	3.0
		Physical property	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	2.5	1.5	3.0
	-5℃	Appearance	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	1.0	1.0	1.0
		Physical property	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0	1.0	1.0	1.0

[0028]

*1: Xanthan gum (product of DAINIPPON PHARMACEUTICAL CO., LTD).

*2: Average polymerization degree: 10; degree of esterification: at least 80%; fatty acid composition: C18:1 and C16; HLB: 2.0.

*3: Fatty acid composition: C18; HLB: 2.1.

*4: Fatty acid composition: C18 and C2; degree of esterification of C18: at least 60%; acetylation rate: at least 35%; HLB: 0.

[0029]

Comparative Examples 1 and 2 relate to mayonnaise incorporating no crystallization inhibitor, and both mayonnaise samples underwent demulsification (oil-off) after storage for 1 month at -5°C and 0°C and caused separation, surface roughness and water separation. Comparative Example 3 relates to mayonnaise in which the polyglycerol fatty acid ester (0.4 % by weight) was used in the oil phase. Sufficient emulsion stability was unable to be achieved in this mayonnaise.

[0030]

On the other hand, when a proper amount of the polyglycerol fatty acid ester (Examples 1 to 3 and 6 to 8), sorbitan fatty acid ester (Example 4) or sucrose fatty acid ester (Example 5) was used as the crystallization inhibitor, no demulsification was caused even after storage for 1

month at -5°C to 5°C, and both appearance and physical property were good.

[0031]

Test Example 2 (Examples 9 to 11):

French dressing (Example 9), Thousand Island dressing (Example 10) and sesame dressing (Example 11) were produced with their corresponding compositions shown in Table 3. More specifically, raw materials for an oil phase were added dropwise to a water phase under stirring to conduct preliminary emulsification. This emulsion was homogenized by a homomixer to obtain the respective dressing samples having an average emulsified droplet diameter of 4 to 12 μm. With respect to the respective dressing samples, the same evaluation as in Test Example 1 was conducted by 6 panelists. As a result, all the dressing samples were good in both appearance and physical property even after storage for 1 month at -5°C to 5°C as shown in Table 3.

[0032]

[Table 3]

(% by weight)

			Example		
			9	10	11
Water phase		Common salt	3.00	2.00	2.50
		Sucrose	5.00	5.00	11.00
		Condiment	0.50	0.50	1.00
		Lemmon juice	2.00	2.00	-
		Thickener *1	0.60	0.40	0.01
		Enzyme-treated yolk	2.00	4.00	-
		Yolk	-	-	1.50
		Tomato ketchup	-	5.00	-
		Pickles	-	4.00	-
		Tomato paste	-	1.00	-
		Miso	-	-	4.00
		Pounded sesame	-	-	7.00
		Soy	-	-	3.00
		5% Brewed vinegar	14.00	14.00	14.00
		Water	32.90	27.10	25.990
Oil phase		Oil Composition 1	19.5	17.0	14.5
		Oil Composition 2	19.5	17.0	14.5
		Polyglycerol fatty acid ester *2	1.0	1.00	1.0
Lyso proportion (%)			40	40	-
Evaluation	5℃	Appearance	3.0	3.0	3.0
		Physical property	3.0	3.0	3.0
	0℃	Appearance	3.0	3.0	3.0
		Physical property	3.0	3.0	3.0
	-5℃	Appearance	3.0	3.0	3.0
		Physical property	3.0	3.0	3.0

[0033]

*1: Xanthan gum (product of DAINIPPON PHARMACEUTICAL CO., LTD).

*2: Average polymerization degree: 10; degree of esterification: at least 80%; fatty acid composition: C18: 1 and C16.

[0034]

[Effects of the Invention]

The acid oil-in-water type emulsion compositions according to the present invention are so excellent in

shelf stability at low temperatures that they do not crack during storage at a low temperature in a refrigerator though they contain DAG at a high concentration, also good in appearance and flavor and useful as diets or lipid metabolism-improving foods.